MEMORANDUM

TO: Mr. Addison Rice
Anderson, Mulholland and Associates

DATE: June 28, 2016

FROM: R. Infante

FILE: 1605350C

RE: Data Validation
Air samples
SDG: 1605350C

SUMMARY

Full validation was performed on the data for one gas sample analyzed for methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on May 17, 2016 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery groups (SDG) 1605350C.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B7AA-1 (05162016)	1605350C-01A	05/17/2016	Air	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results

- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody.

Holding Times and Sample Preservation

All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.

Samples analyzed within method recommended holding time.

Initial and Continuing Calibrations

Methane by ASTM method D-1946 (modified)
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

Laboratory/Field Duplicate Results

Laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL.

LCS/LCSD Results

Methane
LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were not performed (see worksheet).

Calculations were spot checked.

Certification

The following sample 1605350C-01A was analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

Rafael Infante
Chemist License 1888

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Client Sample ID: B7AA-1 (05162016)

Lab ID#: 1605350C-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	**10051906**	**Date of Collection:**	**5/17/16 10:27:00 AM**
Dil. Factor:	**1.73**	**Date of Analysis:**	**5/19/16 10:58 AM**

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00030

Container Type: 6 Liter Summa Canister (100% Certified)

ESTADO LIBRE ASOCIADO DE PUERTO RICO
Rafael Infante Méndez
LIC # 1888
QUIMICO LICENCIADO

FedEx tracking 7831 0105 4196

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Project Manager Terry Taylor
Collected by: (Print and Sign) David Lindstrand
Company Anderson Mulholland Email
Address 2700 Westchester Purchase State NY Zip 10577
Phone 914-251-0400 Fax

Project Info:
P.O. #
Project #
Project Name

Turn Around Time:
☐ Normal
☒ Rush 24-hour
specify

Lab Use Only
Pressurized by:
Date:
Pressurization Gas: N_2 He

Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum Initial (calibrated)	Final (calibrated)	Receipt	Final (psi)
01A	B7AA-1 (05162016)	6L1297	5/17/16	10:27	TO-15	>30	8.0		
	B7AA-1 (05132016)	12695	5/14/16	21:05	Do Not Analyze	730	28.5		

Relinquished by: (signature) Date/Time: David Lindstrand 05/17/16 1500
Received by: (signature) Date/Time: Fed Ex
Relinquished by: (signature) Date/Time:
Received by: (signature) Date/Time: EATL 1045 5/18/16
Relinquished by: (signature) Date/Time:
Received by: (signature) Date/Time:

Notes:

Lab Use Only	Shipper Name	Air Bill #	Temp (°C)	Condition	Custody Seals Intact?	Work Order #
	fedex		N/A		Yes No (None)	1605350

Form 1293 rev.11

DATA REVIEW WORKSHEETS

Project Number:____1605350C_____
Date:_____05/17/2016_____

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from ASTM D-1946 method for measuring permanent gases and light hydrocarbons in refinery and other sources samples using gas chromatography (GC) and a thermal conductivity detector (TCD) and/or flame ionization detection (FID). Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.
The hardcopied (laboratory name) _Eurofins_____ data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: __1605350C_____ Sample matrix: ___Air_____
No. of Samples: _____1_____

Trip blank No.: ____-_____
Field blank No.: _____-_____
Equipment blank No.:___-_____
Field duplicate No.:_____-_____

___X___ Data Completeness
___X___ Holding Times
___N/A_ GC/MS Tuning
___N/A_ Internal Standard Performance
___X___ Blanks
___N/A_ Surrogate Recoveries
___N/A_ Matrix Spike/Matrix Spike Duplicate
___X___ Laboratory Control Spikes
___X___ Field Duplicates
___X___ Calibrations
___X___ Compound Identifications
___X___ Compound Quantitation
___X___ Quantitation Limits

Overall Comments:_Methane_by_ASTM_method_D-1946_(modified)_____
_____

Definition of Qualifiers:
J- Estimated results
U- Compound not detected
R- Rejected data
UJ- Estimated nondetect

Reviewer:_____
Date:__ 06/28/2016_____

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED

All criteria were met _X_
Criteria were not met
and/or see below ____

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH $\leq$ 2, 4°C), no air bubbles.
Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.
Soil samples- 7 days from sample collection.
Cooler temperature (Criteria: 4 $\pm$ 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).
If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)
If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).
If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).
If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).
If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).
If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

All criteria were met __N/A___
Criteria were not met see below _____

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

__N/A_ The BFB performance results were reviewed and found to be within the specified criteria.

__N/A_ BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:
_____

If mass calibration is in error, all associated data are rejected.

Note: Samples analyzed using GC with either TCD or FID detection.

All criteria were met __X__
Criteria were not met
and/or see below ____

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:____01/15/16_____
Dates of continuing calibration:_05/19/16_____
Instrument ID numbers:_____GC-10_____
Matrix/Level:_____Air/low_____

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.				

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.
All %RSD must be ≤ 15 % regardless of method requirements for CCC.
All %Ds must be ≤ 30% regardless of method requirements for CCC.
Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.
If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.
If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).
If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).
If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).
If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).
If any compound has r < 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were met __X__
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
No_field/trip/equipment_blanks_analyzed_with_this_data_package.				

All criteria were met __X__
Criteria were not met
and/or see below _____

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)
ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and ≤ AL, report the compound as not detected (U) at the SQL.
If the concentration is ≥ SQL but ≤ AL, report the compound as not detected (U) at the reported concentration.
If the concentration is ≥ SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately
Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

All criteria were met __N/A___
Criteria were not met
and/or see below _____

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.
List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.
Matrix: solid/aqueous

SAMPLE ID **SURROGATE COMPOUND** **ACTION**

_Surrogate_standards_not_required_by_the_method._____
_____ _____
_____ _____
_____ _____
_____ _____
__ _____

QC Limits* (Air)
_____LL_to_UL_____to_____ ___to_____ ___to_____ ___to_____

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met _____
Criteria were not met
and/or see below __N/A___

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:_____-_____ Matrix/Level:_____-_____

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
_MS/MSD_are_not_required_as_part_of_ASTM-method_D-1946;_blank_spike_used_to_assess_ _accuracy					

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).
If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).
If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met ____
Criteria were not met
and/or see below __N/A___

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:_____ Matrix/Level/Unit:_____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION

Actions:

* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were met _X_
Criteria were not met
and/or see below _____

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.
List the %R of compounds which do not meet the criteria

LCS ID COMPOUND % R QC LIMIT

____LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_recoveries_and_RPD_____
____within_laboratory_control_limits._____

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).
If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.
If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

All criteria were met __X__
Criteria were not met
and/or see below _____

IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample ID_LCS/LCSD_(laboratory_duplicate)____ Matrix:___Air___

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.
Suggested criteria: RPD ± 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for laboratory duplicate (LCS/LCSD) within laboratory control limits.					

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were met __N/A__
Criteria were not met
and/or see below _____

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

* Area of +40% or -40% of the IS area in the associated calibration standard.
* Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION

_Internal_standard_not_required_by_the_method._Samples_quantified_by_external_standard___
_method_____

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were met __X__
Criteria were not met
and/or see below _____

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

LCS 05/19/16

Methane RF = 226379851

[ ] = (2227734265)/(226379851)

= 9.84 % OK

All criteria were met __X__
Criteria were not met
and/or see below _____

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples diluted by a factor of less than 1.73		

B. Percent Solids

List samples which have ≤ 50 % solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)